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[PCD 12: Plastics]

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(पहला पुनरीक्षण)

REAFFIRMED 2005

Indian Standard

MELAMINE-FORMALDEHYDE MOULDING MATERIALS — SPECIFICATION

(*First Revision*)

First Reprint AUGUST 1997

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1994

Price Group 5

Plastics Sectional Committee, PCD 12

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Rated Products Division Council.

Melamine formaldehyde (MF) moulding materials are notablefor their outstanding toughness, excellent electrical insulating properties and water and chemical resistance. Largely because of unlimited colour possibilities and chemical inertness, they are widely used for the manufacture of tableware and electrical appliances.

This standard was originally published in 1966 and was largely based on BS 1322 : 1955 'Specification for aminoplastic moulding materials' issued by the British Standards Institution, which has since been revised after being aligned completely with ISO 2112 : 1977 'Plastic — Aminoplastic moulding materials' issued by the International Organization for Standardization (ISO). This revision has been prepared as a result of review of the standard by Thermosetting Moulding Powders and Resins Subcommittee, PCD 12 : 1, in the light of the present day requirements and advancements that have taken place in this field.

Considerable assistance has been derived from BS 1322 : 1981/ISO 2112 : 1977 issued by the British Standards Institution and ISO 2112 : 1990 published by International Organization for Standardization (ISO), while preparing this revision of the standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 NOVEMBER 1999
TO
IS 3669 : 1994 MELAMINE-FORMALDEHYDE MOULDING MATERIALS — SPECIFICATION

(First Revision)

(*Page* 1, *clause* 2) — Delete 'IS 2221 : 1962' and add 'IS 13360 (Part 3/Sec 4) : 1995/ISO 171 : 1980 Plastics — Methods of testing : Part 3 Physical and dimensional properties, Section 4 Determination of bulk factor of moulding materials' after 'IS 13360 (Part 2/Sec 4) : 1993'.

[*Page* 3, *Table* 1, *Sl No.* (i), *last col*] — Substitute 'IS 13360 (Part 3/Sec 4) : 1995 / ISO 171 : 1980' *for* '5 of IS 2221 : 1962'.

(PCD 12)

Reprography Unit, BIS, New Delhi, India

Indian Standard

MELAMINE-FORMALDEHYDE MOULDING MATERIALS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for various types of melamine-formaldehyde moulding materials which have been further subdivided into different grades based on property levels and requirements.

2 NORMATIVE REFERENCES

The following standards contain provisions, which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
335 : 1983	Insulating oils
2221 : 1962	Methods of test for aminoplastics moulding materials
2259 : 1963	Methods of test for the determination of insulation resistance of solid insulating materials
2584 : 1963	Method of test for electric strength of solid insulating materials at power frequencies
2824 : 1975	Method for determining the comparative tracking index of solid insulating materials under moist conditions (*first revision*)
2828 : 1964	Glossary of terms used in the plastics industry
3389 : 1994	Ureaformaldehyde moulding materials (*first revision*)
4486 : 1967	Recommended methods for the determination of the permitivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelenghts
13360 (Part 1) : 1992	Plastics — Methods of Testing: Part 1 Introduction
13360 (Part 2/Sec 4) : 1993	Plastics — Methods of Testing: Part 2 Sampling and Preparation of Test Specimens Section 4 Preparation of test specimens by machining
13411 : 1992	Glass reinforced polyester dough moulding compounds

3 TERMINOLOGY

For the purpose of this standard, the definitions given under 2 of IS 2221 : 1962 and IS 2828 : 1964 shall apply.

4 TYPES AND GRADES

4.1 Types

There shall be three types of melamine-formaldehyde (MF). moulding materials, classified according to use, as follows:

i) Type MFB — Hot water resistant

Example : Melamine-formaldehyde resin with substantially alpha-cellulose filled materials.

ii) Type MFC — Heat resistant and electrical

Example : Melamine-formaldehyde resin with substantially inorganic filled materials.

iii) Type MFD — Impact resistant

Example : Melamine-formaldehyde resin with chopped cotton-cloth filled materials.

4.1.1 It shall not be inferred from the above that materials of any particular type are necessarily unsuitable for applications other than those indicated, or that specific materials will be suitable for all applications within the wide description given.

4.2 Grades

These types of MF materials have been further sub-divided into following grades based on property levels and property requirements:

i) Grade MF B 10 — Substantially alpha-cellulose-filled material

ii) Grade MF B 12 – Substantially alpha-cellulose-filled material with limited formaldehyde migration for injection-moulding applications

iii) Grade MF B 20 — Substantially wood-flour filled material

5 REQUIREMENTS

5.1 Description

The material shall be homogeneous consisting of uncured or partially cured melamine-formaldehyde resin, combined with fillers and pigments or dyes, as required.

5.2 The materials complying with this specification shall meet the appropriate property requirements shown in Table 1.

6 TEST SPECIMENS

Bulk factor and flow shall be determined on the moulding material. The other properties shall be determined on moulded test specimens prepared in accordance with Annex A of IS 3389 : 1994 with the following moulding conditions:

Moulding Materials	*Moulding Temperature*	*Moulding Pressure*	*Curing Time per mm of Thickness*
Melamine materials with cellulose, wood flour or mineral fillers	150-160°C	15-35 MPa	30-60 seconds
Melamine materials with coarse fibrous fillers	150-160°C	30-50 MPa	30-60 seconds

If drying or preheating is carried out, the method, time and temperature shall be stated. In case of preheating, a curing time of 20-30 seconds per millimetre of thickness is permissible.

It is permissible to machine test specimens [*see* IS 13360 (Part 2/Sec 4) : 1993] from sheet moulded in accordance with the above moulding conditions as long as it can be shown that the specimens give result which do not differ significantly from those obtained using moulded specimens.

Test specimens to be used for determining the properties given in Table 1 shall be conditioned under prevailing atmospheric conditions as allowed in IS 13360 (Part 1) : 1992, unless otherwise stated in the method of test or agreed on between the interested parties.

Test shall commence not less than 16 h and not more than 72 h after the test specimens have been moulded, unless otherwise specified in the test methods.

When the test specimens have been moulded from powder that has been preheated or dried, then this fact shall be stated in the test report. The conditions of preheating or drying shall also be given.

7 METHODS OF TEST

For mechanical tests, it is permissible for the forces to be applied in an alternative direction to that specified in the test methods, provided that the results do not differ significantly from those obtained when the force is applied in the specified direction.

7.1 Determination of Flexural Stress at Rupture

Fire test specimens of length not less than 80 mm, width 10 mm and of thickness 4 mm shall be used.

For both moulded bars and test specimens machined from material moulded in the form of a plate, the load shall be applied parallel to the direction of moulding pressure, the test speed shall be 2 mm/min ± 0·2 mm/min. Test shall be carried out in accordance with Annex F of IS 13411 : 1992.

7.2 Determination of Izod Impact Strength

Five test specimens shall be used. In the case of specimens machined from material moulded in the form of a plate, the load shall be applied perpendicular to the direction of the moulding pressure.

Test shall be carried out in accordance with Annex E of IS 13411 : 1992 (Method 2A).

Table 1 Requirements for Melamine-Formaldehyde Moulding Material

(*Clause* 5.2)

Sl No.	Property	Units	Type MFB			Type MFC	Type MFD	Methods of Test
			Grade MF B10	Grade MF B12	Grade MF B20	Grade MF C10	Grade MF D10	
A)	*Properties Measured on Moulding Powder*							
i)	Bulk factor, *Max*	—	←—Limits to be agreed on between the interested parties—→					5 of IS 2221 : 1962
ii)	Flow	—	—do—					To be agreed between the interested parties
B)	*Properties Measured on Test Specimens*							
i)	Flexural stress at rupture, *Min*	MPa	70	70	60	50	50	Annex F of IS 13411 : 1992
ii)	Izod impact strength, *Min*	KJ/m^2	1·5	1·5	1·5	1·5	6·0	Annex E of IS 13411 : 1992 (Method 2A)
iii)	Temperature of deflection under load, *Min*	°C	150	140	130	140	120	Annex H of IS 13411 : 1992
iv)	Incandescence resistance	—	←—Limits to be agreed on between the interested parties—→					Annex C of IS 3389 : 1994
v)	Insulation resistance after 24 h in water, *Min*	ohm	1×10^{10}	1×10^{10}	1×10^{8}	1×10^{8}	1×10^{8}	IS 2259 : 1963
vi)	Electric strength, *Min*	MV/m	4·0	3·0	3·0	3·0	2·0	IS 2584 : 1963
vii)	Comparative tracking resistance (CTI), *Min*	V	CTI 500	CTI 500	CTI 500	CTI 500	CTI 500	IS 2824 : 1975
viii)	Dielectric dissipation factor (Tan δ), 1 MHZ, *Max*	—	—	—	—	0·30	—	IS 4486 : 1967
ix)	Water absorption, *Max*							
	a) Hot	mg	130	130	200	100	—	Annex D of IS 13411 : 1992 (Method 4)
	b) Cold	mg	100	100	150	80	120	—do— (Method 1)
x)	Mould shrinkage, *Max*	%	0·9	1·1	0·9	0·7	0·8	Annex B of IS 13411 : 1992
xi)	Post-moulding shrinkage, 48 h *Max*	%	←—Limits to be agreed on between the interested parties—→					—do—
xii)	Extractable formaldehyde, *Max*							
	a) Water	μg/cm^2 of specimen surface	—	To be reported	—		—	Annex A
	b) Acetic acid	—do—	—	—do—	—	—	—	—do—
	c) Alcohol	—do—	—	—do—	—	—	—	—do—

NOTE — The values specified in this table represent the mean result for the property measured except for extractable formaldehyde, in which case the individal results shall comply with the limit.

7.3 Determination of Temperature of Deflection Underload

Two test specimens of length not less than 110 mm, width 10 mm and thicknes 4 mm shall be used.

Test shall be carried out in accordance with Annex H of IS 13411 : 1992 (Method A).

7.4 Determination of Incandescence Resistance

Five specimens each measuring 10 mm × 4 mm × 80-130 mm in length shall be used for the determination. Test shall be carried out in accordance with Annex C of IS 3389 : 1994.

After 3 minutes turn the ignition bar holder away from the specimen and note whether there is any visible flame on specimen during the next 30 seconds.

7.5 Determination of Insulation Resistance

At least two specimens shall be used. Each test specimen shall be in the form of a flat plate moulded to a thickness of 3·0 mm ± 0·25 mm. The taper pin electrodes shall be used. Before carrying out the test, specimen shall be conditioned (without electrodes) in an oven at 50 ± 2°C for 24 ± 1 h and then cooled to room temperature in a desiccator. It shall then be immersed in distilled or deionized water at 27 ± 2°C for 24 ± 1 h. Before testing, the surface water shall be removed with blotting or filter paper or with a clean, absorbent cloth, and the electrodes fitted. The measurement of insulation resistance shall be made within 5 min after the end of the immersion.

Test shall be carried out in accordance with IS 2259 : 1963.

7.6 Determination of Electric Strength at Power Frequencies

At least two test specimens shall be used for the determination. Each specimen shall be 3·0 ± 0·25 mm thick and not less than 100 mm in diameter. Each test specimen shall be immersed in oil at a temperature of 90 ± 2°C for 15 to 20 min before the test and during the test. The oil shall preferably be one complying with the requirements of IS 335 : 1983. The 20 s step-by-step method shall be used.

Test shall be carried out in accordance with IS 2584 : 1963.

NOTE — It may be necessary to measure the short-term value on an additional test specimen in order to determine the initial voltage to be applied.

7.7 Determination of Tracking Resistance Under Moist Conditions

Test shall be carried out in accordance with IS 2824 : 1975 Proof test. The applied voltage shall be the numerical value of the comparative tracking index given in Table 1. Two determinations shall be made. The material shall not track before 51 drops of electrolytes have fallen.

7.8 Determination of Dielectric Dissipation Factor

Two test specimens shall be used.

The conditions of test shall be:

Frequency	1 MHz
Temperature	27 ± 2°C
Relative humidity	65 ± 5 %; and

electrodes metal foil or conductive paint.

Test shall be carried out in accordance with IS 4486 : 1967.

7.9 Determination of Water Absorption

Two test specimen 50 ± 1 mm in diameter and 3 ± 0·2 mm thick shall preferably be used. As an alternative, when agreed between the purchaser and the supplier, square specimens of side 50 ± 1 mm cut from 4 ± 0·2 mm thick moulded plates may be used. If this alternative type of test specimens is used, the requirements shall also be the subject of agreement between the purchaser and the supplier.

7.9.1 *Hot Water Absorption*

Test shall be carried out in accordance with Annex D of IS 13411 : 1992 (Method 4).

7.9.2 *Cold Water Absorption*

Test shall be carried out in accordance with Annex D of IS 13411 : 1992 (Method 1).

7.10 Determination of Mould Shrinkage

Two test specimens shall be used. For material intended for injection or transfer moulding, the method of preparation of test specimens shall be agreed between the purchaser and the supplier.

Test shall be carried out in accordance with Annex B of IS 13411 : 1992.

7.11 Determination of Post-Moulding Shrinkage

Two test specimens shall be used. For materials intended for injection or transfer moulding, the method of preparation of the test specimens shall be agreed between the purchaser and the supplier.

Test shall be carried out in accordance with Annex B of IS 13411 : 1992.

7.12 Determination of Extractability of Formaldehyde

Two test specimens shall be used.

Test shall be carried out in accordance with Annex A of this standard.

8 FREQUENCY OF TEST

In determining compliance of a material with the requirements of this standard, it is usually appropriate to determine some properties more frequently than others. For example, if the properties listed below for frequent determination are determined on one batch in ten batches, it may be sufficient to determine the remaining properties on one batch in fifty batches.

a) *Properties requiring frequent determination*

   Bulk factor
   Flow
   Flexural stress at rupture
   Insulation resistance after 24 h in water
   Dielectric dissipation factor
   Water absorption, cold
   Mould shrinkage
   Extractable formaldehyde

b) *Properties requiring less frequent determination*

  Impact of strength, Izod
  Temperature of deflection under load
  Incandescence resistance
  Electric strength
  Comparative tracking resistance
  Water absorption, hot
  Post-shrinkage, 48 h

9 PACKING AND MARKING

9.1 Packing

The material shall be packed in well-closed containers or as agreed to between the purchaser and the supplier.

9.2 Marking

The containers shall be securely closed and legibly marked with the following information:

a) Indication of the source of manufacture and recognized trade-mark, if any;

b) Name, type and grade of the material;

c) Colour of the material;

d) Net mass of the material in the container;

e) Month and year of manufacture; and

f) Batch No. or Code No.

9.3 BIS Certification Marking

The container may also be marked with the STANDARD MARK.

9.3.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

10 SAMPLING

10.1 General

In drawing, preparing, storing and handling of samples, the following precautions and directions shall be observed:

a) Samples shall be taken in an exposed place.

b) The sampling instrument shall be made of stainless steel and shall be clean and dry.

c) Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for sample from adventitions contamination.

d) Samples shall be placed in suitable, clean, dry, air-tight metal or polythene containers on which the material has no action.

e) Sample containers shall be of such a size that they are almost completely filled by the sample.

f) Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, the name of the material, etc.

g) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the room temperature (cool, dry and away from sun).

10.2 Scale of Sampling

10.2.1 *Lot*

All the containers of melamine formaldehyde moulding material of the same type and grade, manufactured under similar conditions of production shall be grouped together to consititute a lot.

10.2.1.1 *Number of samples*

For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out on each lot separately. The number of containers to be selected from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling

Lot Size	No of Containers to be Selected from the Lot
(1)	(2)
Up to 150	3
151 to 300	4
301 to 500	5
501 and above	7

Each container in the sample shall be selected at random from each lot. For this purpose, reference may be made to IS 4905 : 1968 'Methods of random sampling'.

10.3 Preparation of Test Samples and Referee Sample

10.3.1 *Sampling Instrument*

For sampling, instrument made of stainless steel shall be as used as shown in Fig. 1. It shall be capable of taking samples from all points when inserted in the container.

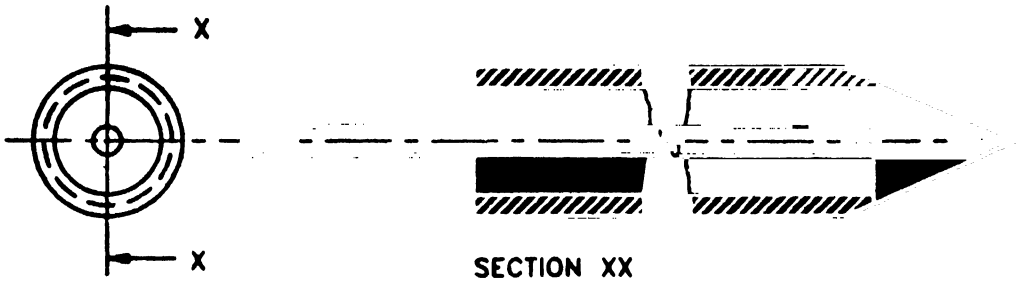

FIG. 1 SAMPLING INSTRUMENT

10.3.2 From each of the containers selected, representative portion of the material shall be drawn with the help of the sampling instrument. For this purpose, the material shall be taken from different points at least 75 mm away from any surface in the case of large containers, and 25 mm away in the case of small containers. The total quantity of the material collected from each container shall be sufficient for determinations of the various characteristic as specified in the relevant material specification (*see* **10.3.3** and **10.3.4**).

10.3.3 Out of these portions, a smal! but equal quantity of the material shall be taken out and mixed thoroughly to form composite test sample. The composite test sample thus formed shall be divided into three parts and transferred to separate bottles. One of these bottles shall be marked for the purchaser, another for the supplier and the third for the referee.

10.3.4 The remaining portion of the material from each container shall be divided into three parts and each such part shall be transferred to separate bottles which are then sealed airtight with stoppers and labelled with full identification particulars of the sample. The material in each sealed bottle shall constitute an individual test sample. These individual test samples shall be grouped into three sets in such a way that every set has a test sample representing each container selected. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

10.3.5 *Referee Sample*

Referee sample shall consist of the composite test sample and a set of individual test samples marked for the purpose and shall bear the seals of the purchaser and the supplier and shall be used in the case of a dispute between the two.

10.4 Number of Tests

The test for description (5.1), Bulk factor [Table 1 (i)] and Flow [Table 1 (ii)] shall be made on each of the individual samples while tests for all other characteristics shall be made on the composite sample.

10.5 Criteria for Conformity

10.5.1 *Individual Samples*

10.5.1.1 From test results for each characteristic tested on individual samples, the Average ($\bar{X}$) and Range (R) (if sample size is less than 10) or Average Range ($\bar{R}$) (if sample is greater than or equal to 10), shall then be calculated from test results.

$$\text{Average} (\bar{X}) = \frac{\text{Sum of all test results}}{\text{Number of test results}}$$

Range (R) = Difference between maximum and minimum test results

NOTE — For sample size 10 or more, range is calculated for five test results taken in order in which they are obtained and then average Range ($\bar{R}$) is computed.

The lot shall be considered as meeting the requirements of these characteristics, if

> ($\bar{X} + 0{\cdot}6\,R$) or ($\bar{X} + 0{\cdot}6\,\bar{R}$) is less than or equal to the maximum specified limit or ($\bar{X} - 0{\cdot}6\,R$) or ($\bar{X} - 0{\cdot}6\,\bar{R}$) is greater than or equal to the minimum specified limit for each of these characteristics.

10.5.2 *Composite Sample*

10.5.2.1 In respect of each charateristic tested on the composite sample, the lot shall be declared as conforming to the requirements of the specification if the test results on the composite sample satisfy the corrcsponding requirements.

10.5.2.2 The lot shall be declared as conforming to the requirements of the specification if **10.5.1** and **10.5.2** are satisfied.

ANNEX A

(*Clauses* 7.11)

DETERMINATION OF EXTRACTABILITY OF FORMALDEHYDE

(Adopted from ISO 4614 : 1977)

A-1 PRINCIPLE

Certain liquids, simulating common food and beverage constituents, are placed in contact with mouldings of the sample material, under defined conditions. The formaldehyde content of the liquid is then determined and the quantity of formaldehyde extracted per unit area of contact with the moulding is calculated.

Two procedures for the determination of formaldehyde in the liquid are given.

A-2 TEST SPECIMENS

A-2.1 Form

Moulded containers, for example beakers or cups, having an internal surface area of 150 to 250 cm² and capacity 150 to 250 cm³ are suitable as test specimens. The quotient of the number expressing the surface wetted in square centimetres by that expressing the volume of liquid in cubic centimetres must lie between 0·75 and 1.

A-2.2 Number

Six specimens are required. The determination is carried out in duplicate with each of the three extraction liquids (A-3).

A-3 EXTRACTION LIQUIDS

A-3.1 Water, distilled or de-ionized.

A-3.2 Acetic Acid

30 g/l solution of glacial acetic acid in distilled water.

A-3.3 Ethanol

100 g/l solution of ethanol in distilled water.

A-4 EXTRACTION PROCEDURE

Rinse the test container with warm distilled water and dry thoroughly.

Place a suitable quantity of the extraction liquid at 80°C into the test container at room temperature. Cover the container with a watch glass to protect against evaporation and contamination, and allow to stand in air at room temperature for 30 min.

Transfer the extract (without washing) to a 250-ml conical flask, stopper the flask and cool the solution to 27 ± 2°C. Immediately carry out the formaldehyde analysis by Procedure A or Procedure B.

A-5 DETERMINATION OF FORMALDEHYDE — PROCEDURE A

A-5.1 Reagents

All reagents, including distilled water and extraction liquids, shall be free from formaldeyde in amounts detectable by the method described.

A-5.1.1 Water distilled or de-ionized.

A-5.1.2 *Chromotropic Acid Disodium Salt* (disodium 4,5-dihydroxy 2,7-naphthalene sulphonate) solution.

Dissolve 0·50 g of chromotropic acid disodium salt in 50 ml of distilled water. Transfer the solution to 100 ml volumetric flask and make up to volume. Prepare the fresh solution each day.

A-5.1.3 *Formaldehyde Solutions*

All solutions shall be made up at 27 ±2°C.

A-5.1.3.1 *Formaldehyde stock solutions*

Pipette 25·0 ml of industrial formalin (containing approximately 400 g/l formaldehyde) into a 1 000-ml volumetric flask and make up to volume with distilled water. Determine the concentrations of formaldehyde in the stock solution using the standard procedure.

This solution shall not be kept longer than one week.

A-5.1.3.2 *Formaldehyde working solution A*

Pipette 10·0 ml of formaldehyde stock solution (**A-5.1.3.1**) into a 1 000-ml volumetric flask and make up to volume with the extraction liquid to be used. Mix thoroughly.

A-5.1.3.3 *Formaldehyde working solution B* (approximately 10 mg/l).

Pipette 10·0 ml of solution A (**A-5.1.3.2**) into a 100-ml volumetric flask and make up to the mark with the extraction liquid to be used.

NOTE — The exact concentration of formaldehyde in solution B can be calculated from the known formaldehyde content of the stock solution (A-5.1.3.1).

Solutions A and B shall be made up immediately prior to use.

A-5.1.4 Sulphuric Acid — concentrated, 81 % (*m*/*m*) of analytical reagent grade.

A-5.2 Apparatus

A-5.2.1 *Test Tubes* — fitted with ground glass stoppers.

A-5.2.2 Water bath at 60°C.

A-5.2.3 *Visible Region Spectrophotometer* — fitted with 10 mm path length glass cells.

A-5.2.4 *Volumetric Glassware*

A-5.2.4.1 Pipette, 1, 10 and 25 ml.

A-5.2.4.2 Two burettes, 5 ml.

A-5.2.4.3 Burette, 50 ml.

A-5.2.4.4 Graduated flasks, 100- and 1000-ml.

A-5.3 Procedure

Carry out the procedure specified in **A-5.3.1** and **A-5.3.2** for each extraction liquid.

A-5.3.1 *Establishment of the Calibration Curve*

To six separate test tubes using the 5 ml burettes add the volumes of solution B shown in the following table and make each solution up to 1·0 ml using the appropriate amount of the extraction liquid.

Solution B, ml	1·0	0·8	0·6	0·4	0·2	0·1
Extraction liquid, ml	0	0·2	0·4	0·6	0·8	0·9

This will give the equivalent of solutions containing from approximately 10 to 1 μg/ml formaldehyde. Into each of the test tubes pipette 1·0 ml of chromotropic acid disodium salt solution and add slowly, while shaking 8·0 ml of sulphuric acid from the 50 ml burette. Mix thoroughly by shaking and stopper the tubes. Place the tubes in the water bath at 60°C for 30 min. Remove the tube from the water bath and allow them to stand at room temperature for 45 to 60 min (solutions C).

Transfer a portion of each solution C, in turn, to a 10 mm glass cell and measure its absorbance at 570 nm against distilled water.

Carry out a blank determination on the reagents alone using 1·0 ml of the extraction liquid in place of the formaldehyde solution.

Plot a graph of absorbance (10 mm cell) as ordinate against concentration of formaldehyde (μg/ml) as abscissa. This shall be a straight line passing through the origin of coordinates.

A-5.3.2 *Determination of Formaldehyde*

Pipette 1·0 ml of extract and 1·0 ml of chromotropic acid disodium salt solution into a test tube and add slowly, while shaking 8·0 ml of sulphuric acid from the burette. Mix thorougly by shaking and stopper the tube. Place the tube in the water bath at 60°C for 30 min. Remove the tube from the water bath and allow it to stand at room temperature for 45 to 60 min (solution C).

Transfer a portion of solution C to a 10 mm glass cell and measure its absorbance at 570 nm against distilled water.

Prepare a blank by treating 1·0 ml of the extraction liquid in exactly the same manner as the extract.

Read off the concentration of formaldehyde in the extract from the calibration curve.

NOTE — The procedure requires that the formaldehyde content of the extracts shall be in the concentration range 1·0 to 10·0 μg/ml. If the formaldehyde content of the extract is found on investigation to be greater than 10 μg/ml, then the extract shall be diluted, and a 1 ml aliquot of this diluted extract used for the determination.

A-6 DETERMINATION OF FORMALDEHYDE — PROCEDURE B

A-6.1 Reagents

All reagents, including distilled water and extraction liquids, shall be free from formaldehyde in amounts detectable by the method described.

A-6.1.1 *Water*, distilled or de-ionized.

A-6.1.2 *Acetylacetone*, solution.

Dissolve 150 g of ammonium acetate in distilled water and add 3·0 ml of glacial acetic acid and 2·0 ml of acetylacetone to this solution. Transfer the solution to a 1 000-ml volumetric flask and make up to volume with distilled water. Prepare the fresh solution each day.

A-6.1.3 *Formaldehyde*, solutions.

All solutions shall be made up at 27 ± 2°C.

A-6.1.3.1 *Formaldehyde stock solution*

Pipette 25·0 ml of industrial formalin (containing approximately 400 g/l formaldehyde) into a 1 000-ml volumetric flask and make up to volume with distilled water. Determine the concentration of formaldehyde in the stock solution using the standard procedure.

This solution shall not be kept longer than one week.

A-6.1.3.2 *Formaldehyde working solution A*

Pipette 10·0 ml of formaldehyde stock solution (**A-6.1.3.1**) into a 1 000-ml volumetric flask and make up to volume with the extraction liquid to be used. Mix thoroughly.

A-6.1.3.3 *Formaldehyde working solution B (approximately 10 mg/l)*.

Pipette 10·0 ml of solution A (**A-6.1.3.2**) into a 100-ml volumetric flask and make up to the mark with the extraction liquid to be used.

NOTE — The exact concentration of formaldehyde in solution B can be calculated from the known formaldehyde content of the stock solution (**A-6.1.3.1**).

Solutions A and B shall be made up immediately prior to use.

A-6.2 Apparatus

A-6.2.1 *Test Tubes*—fitted with ground glass stoppers.

A-6.2.2 *Water Bath* — 60°C.

A-6.2.3 *Visible Region Spectrophotometer* — fitted with 10 mm path length glass cells.

A-6.2.4 *Volumetric Glassware*

A-6.2.4.1 Pipettes, 5, 10 and 25 ml.

A-6.2.4.2 Two burretes, 5 ml.

A-6.2.4.3 Burette, 50 ml.

A-6.2.4.4 Graduated flasks, 100 and 1 000-ml.

A-6.3 Procedure

Carry out the procedure specified in **A-6.3.1** and **A-6.3.2** for each extraction liquid.

A-6.3.1 *Establishment of the Calibration Curve*

To six separate test tubes using the 5 ml burette add the volumes of solution B shown in the following table and make each solution up to 5·0 ml using the appropriate amount of the extraction liquid.

Solution B, ml	4·0	3·0	2·0	1·0	0·5	0·25
Extraction liquid, ml	1·0	2·0	3·0	4·0	4·5	4·75

This will give the equivalent of solutions containing from approximately 8 to 0·5 μg/ml formaldehyde. Into each of the test tubes pipette 5 ml of acetylacetone solution. Mix thoroughly by shaking and stopper the tubes. Place the tubes in the water bath at 60°C for 20 min. Remove the tubes from the water bath and allow them to stand at room temperature for 45 to 60 min (solution C).

Transfer a portion of each solution C, in turn, to a 10-mm glass cell and measure its absorbance at 415 nm against distilled water.

Carry out a blank determination on the reagents alone using 5·0 ml of the extraction liquid in place of the formaldehyde solution.

Plot a graph of absorbance (10 mm cell) as ordinate against concentration of formaldehyde (μg/ml) as abscissa. This shall be a straight line passing through the origin of coordinates.

A-6.3.2 *Determination of formaldehyde*

Pipette 5·0 ml of extract and 5·0 ml of acetylacetone solution into a test tube. Mix thoroughly by shaking and stopper the tube. Place the tube in the water bath and allow it to stand at room temperature for 45 to 60 min (solution C).

Transfer a portion of solution C to a 10-mm glass cell and measure its absorbance at 415 nm against distilled water.

Prepare a blank by treating 5·0 ml of the extraction liquid in exactly the same manner as the extract.

Read off the concentration of formaldehyde in the extract from the calibration curve.

NOTE — The procedure requires that the formaldehyde content of the extracts shall be in the concentration range 0·5 to 8·0 μg/ml. If the formaldehyde content of the extract is found on investigation to be greater than 8 μg/ml, then the extract shall be diluted, and a 5-ml aliquot of this diluted extract used for the determination.

A-7 EXPRESSION OF RESULTS

From the absorbance of the extract solution, deduce the formaldehyde content of the original extract.

Then:

1) formaldehyde extracted (μg/ml of extraction liquid) $= a \times f$

2) formaldehyde extracted (μg/cm² of specimen surface) $= \frac{a \times V \times f}{A}$

where

a is the formaldehyde content, in micrograms per millilitre, of the test solution;

f is the dilution factor (if required by the terms of the note in **A-5.3.2** and **A-6.3.2**);

V is the total volume, in millilitres, of extraction liquid used;

A is the area of specimen in contact with extraction liquid, in square centimetres.

For each extraction liquid calculate the individual test results for the two determinations.

A-8 TEST REPORT

The test report shall include the following particulars:

i) complete identification of the product tested;

ii) description of test specimen, design, capacity and ratio of wetted surface to volume of liquid;

iii) moulding conditions, curing time and temperature;

iv) statement of which test procedure has been used (A or B);

v) the individual test results.

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